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Iron metallurgy of the Xianbei period in Tuva (Southern Siberia)

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ABSTRACT

We present results of the complex investigation of large-scale iron production at the site of Katylyg 5 (Tuva, Southern Siberia) dating to 3rd-4th c. AD. The excavations have uncovered nine trapezoid underground smelting furnaces, a tonne of smelting slag, smithing remains and a charcoal production zone. The investigation of slag by Optical microscopy, SEM-EDS and ICP-MS confirms the performance of smelting and smithing operations at the site, and also suggests that the smelted ore was magnetite, associated with quartz. The presence of copper (bronze) prills in most of the smithing slag indicates that copper was worked alongside iron in the smithing hearths. The spatial division of the site into three different production zones (smelting, smithing and charcoal-production) suggests a well-organized and self-sufficient industry, that was probably tightly controlled throughout all stages of the chaîne opératoire. The trapezoid furnaces identified at Katylyg, are also known from Cis-Baikal region where they date from the end of the 1st millennium BCE and throughout most of the 1st millennium AD. This suggests that the technology of trapezoid furnaces, along with the Kokel culture to which they are attributed, likely emerged in Tuva with the migrations from the Baikal region due to the westward Xianbei expansion during 1st-3rd c. AD.

1. Introduction

Over several millennia, Altai-Sayan region has attracted nomadic people not only by the good pastures but also by the abundant resources of copper, iron and gold ores (Sunchugashev, 1969, 1979; Zinyakov, 1988; Konstantinov et al., 2018). Despite their presence, the adoption of different metallurgical technologies did not happen simultaneously. If the tradition of copper metallurgy goes back to the Afanasievo culture (3300–2500 BCE), the exploitation of gold probably started only during the Iron Age (800–300 BCE), while iron production technology may have been adopted even after the Iron Age (Chernykh, 2010; Zaikov et al., 2016; Zavyalov and Terekhova, 2015).

Finds of iron objects, including tools, horse fittings and weaponry in Sayano-Altai, are already attested for Iron Age burial complexes of the 7th-3rd c. BCE (Gryaznov, 1950; Kubarev and Shulga, 2007; Chugunov et al., 2017). However, iron did not fully replace bronze in the named types of objects. Furthermore, no smelting furnaces can be securely identified to the period before the 3rd c. BCE in the region, although this identification is challenging by itself, as the only means of dating these metallurgical installations are the typology of the associated cultural remains (Sunchugashev, 1969).

This general lack of production residues in Siberia, as well as the use of advanced iron-making techniques (cementation, the use of steel, copper gilding of the iron blade) in some of the Iron Age objects found there, have prompted researchers to propose a non-local origin for the iron objects from Siberia in the absence of indigenous iron production during the Iron Age. It was suggested that iron was imported to Siberia from Iran, Central Asia, or Xinjiang (Zavyalov and Terekhova, 2015, Chlenova, 1992: 222; Guo, 2009). According to the other scholars (Brosseder, 2015; Linduff and Rubinson, 2010), this influx of various luxury goods into the region could have been promoted by the developed trade networks, and the major socio-cultural changes during the Iron Age. Furthermore, in some regions including Tuva, the demand for iron could have been compensated by the developed metal technologies relying on the production of tin-bronzes or arsenical copper (Park et al., 2020a, Tishkin et al., 2014, Havrin, 2007).

The new research therefore is changing the paradigm of the time of full adoption of iron technology in Southern Siberia. According to recent

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studies, this happens during the late 1st c. BC – early 1st c. AD (Amzarakov, 2015; Zavyalov and Terekhova, 2015; Vodyasov and Zaitceva, 2020), and is probably associated with the rise of the Xiongnu empire. This powerful nomadic entity was capable of developing/adopting and spreading their own bloomery smelting and smithing technologies, different from those used by the Han China, across the wide area of the eastern steppes (Park et al., 2010; 2020b, Sasada and Chunag, 2014; Ishtseren, 2015; Zavyalov and Terekhova, 2015; Wensuo, 2016).

Despite the apparent role of the Xiongnu in the spread of iron technology, the broader character and organization of metallurgical production among the Xiongnu is still poorly understood. This is partly due to the fact that most of the available archaeometallurgical evidence is finished metal objects, while the production remains are often lacking or insufficiently studied (Brosseder, 2015: 206, Linduff and Rubinson, 2010, Miller and Brosseder, 2013, Khavrin, 2011). Nonetheless, these remains appearing as early as the Xiongnu period, are still attested in South Siberia, including Sayan, Altai, Tuva and Baikal, and Mongolia, which emphasizes the prominent role of these regions in production and supply of metal to the Xiongnu (Kradin, 2011: 83, Vodyasov and Zaitceva, 2020, Davydova, 1995, Sasada and Chunag, 2014, Kharinsky, 2014).

The earliest iron production remains from the named regions are often found inside the underground smelting installations dug in clay soil suggesting that this type of smelting furnace was a common trait of the Xiongnu iron smelting tradition, perhaps continuing into the subsequent Xianbei era (Sunchugashev, 1969, 1979, Kharinsky and Snopkov, 2004, Sasada and Chunag, 2014, Pleiner, 2000: 188). Beyond this, the lack of systematic research on production residues and the non-performance of material analyses leaves most of the questions about the scale, organization, specialization, sources of raw materials of nomadic metallurgical industries unresolved.

Previously, the density of archaeometallurgical research was particularly uneven, and often insufficient, in the Altay-Sayan region, including Tuva. After the intensive surveying and excavations of metallurgical sites during 1960-1970′s (Sunchugashev, 1969, 1979; Zinyakov, 1988), the interest in this topic was abandoned. Only during the last decade, the archaeological work in Altay-Sayan region resumed, the existing chronologies of iron production were refined and discussions about the origin and development of iron metallurgy there continued (Amzarakov, 2015; Murakami et al., 2019; Vodyasov et al., 2020; Vodyasov and Zaitceva, 2020, Sadykov, 2015, 2018a, 2018b).

Despite the revival of the interest in the ancient metallurgy of Sayano-Altay, the archaeology of the Tuva region largely remained obscure. The lack of radiocarbon dating programs of metallurgical sites significantly complicated the understanding of their chronology and the general timing of adoption of iron metallurgy (Tulush, 2017). However, the complexity and the long-lasting character of local iron-making traditions, ranging from the Xiongnu era and into the following periods of Xianbei, Uygur and Mongolian rule, is suggested from the existence of the different typologies of furnaces, and the presence of characteristic cultural remains (e.g., pottery) at the site attesting to different chronological periods (Sunchugashev, 1969: 107, 111, Sadykov, 2018b).

The present study, therefore, focuses on the complex investigation of iron metallurgy in the Xianbei period (1st-3rd c. AD) at the settlement of Katylyg 5 in the central Tuva (Sadykov, 2015, 2018a). We combine the data obtained during field excavations, radiocarbon dating, and the material analyses of metallurgical remains in order to broadly characterize the iron production at the site including typology of furnaces, performed operations, ore sources, scale, organization, chronological parallels and the origins of the iron-making tradition within the social context of Siberia and the eastern steppe and forest-steppe regions.

2. Metallurgy at Katylyg 5

The settlement of Katylyg 5 (hereafter Katylyg) is located 30 km to the north-west from Kyzyl, on the right bank of the Eerbek River (right tributary of Yenisey) in forest zone (Fig. 1). The settlement of ca. 4000 m2 in total, is located at the edge of the natural terrace, limited by the inner and outer ramparts separated by two parallel ditches between them (Fig. 2). The overall width of the defence line comprises ca. 12 m. The site was first discovered by Sadykov in 2012, who uncovered more than 2000 m^2 area (Sadykov, 2015, 2018a).

Katylyg, despite its fortifications, does not contain traces of habitation areas, which may be due to the seasonal (summer) use of the site, or its unique purpose as a production centre. The material assemblages from the site include pottery, bone-made jewellery, stone spindle whorls, iron knives, awls, bone and iron arrowheads, as well as abundant remains of iron production including: slags (both massive blocks inside the undergoing furnaces and fragments scattered throughout the cultural layer); charcoal, fired/slagged clay lining and clay bricks; clay tuyeres and the walls of smithing hearth with tuyere holes.

Based on ca. 8000 pottery finds (Fig. 3), Katylyg belongs to Kokel culture and is currently the only known Kokel settlement. The Kokel culture, limited by the modern borders of Tuva, was initially identified as Xiongnu-influenced, originating from the arrival of Xiongnu in the region (2nd c. AD). Subsequently, the lower chronological boundary of the Kokel culture was refined as 2nd-3rd c. AD, based on the radiocarbon dates obtained from two sites (Sadykov, 2018a; Milella et al., 2021), allowing the abandonment of the hypothesis about its Xiongnu cultural origins.

The emergence of the Kokel culture, therefore, should be regarded in the context of the following Xianbei era (Savinov, 2010; Khudyakov, 2019), although no direct evidence for the existence of Xianbei in Tuva, except the finds of typical jewellery, was identified so far (Khudyakov et al., 1999). Nevertheless, the finds of the Xianbei-like armament, a sharp change in the ceramic tradition and the anthropological type of the Tuva population suggest that the Kokel culture emerged due to the westward human migrations, somehow related to the process of a Xianbei expansion during 1st-3rd c. AD (Sadykov, 2017, 2018a).

2.1. *Smelting zone and the typology of the furnaces*

Spatial analysis of the distribution of material remains at the site reveals three different production zones: (1) north-western zone for smelting containing remains of smelting furnaces and smelting slag; (2) south-eastern zone for smithing containing clay tuyeres and fragments of smithing hearths; (3) south-western zone with pits for preparation of charcoal (Fig. 2).

The excavations undertaken in the north-western zone of the site revealed nine smelting furnaces, all characterized by almost identical typology and dimensions (Fig. 4). The absence of any remains of furnace superstructures (from clay or stone) suggests that the furnaces should be classified as an underground-type (Pleiner, 2000: 188-189).

The shape and dimensions of smelting chambers can be deduced from the well-preserved large slag blocks that remained in situ inside each chamber. Based on the shape of the slag blocks, 7 out of 9 furnaces are trapezoid in plan, narrowing towards the bottom where the underground channel was located. Only two furnaces (No. 1, 6) slightly differ from the rest due to rectangular shape design. The typical depth of the smelting chamber is ca. 1 m, with the slag blocks filling the lower part of the furnace up to the height of 0.5–0.6 m. The length of sidewalls of the furnaces (in plan) ranges: 50–60 cm, the width of rear walls: 55–55 cm, the width of front walls: 18–30 cm (Figs. 4–6). Almost all furnaces have inclined rear walls.

Apart from the slag blocks that remained inside each furnace, some amount of tapped slag was also found in situ near the furnaces, and sometimes inside the underground channels (Figs. 4A–B, 5). The tapping of this slag likely happened towards the end of the smelt, when the smelting chamber was full of slag to the acceptable level, while the ore charging and smelting process continued. Only one out of nine furnaces (No 6) did not preserve the tapped slag, possibly due to the fact that

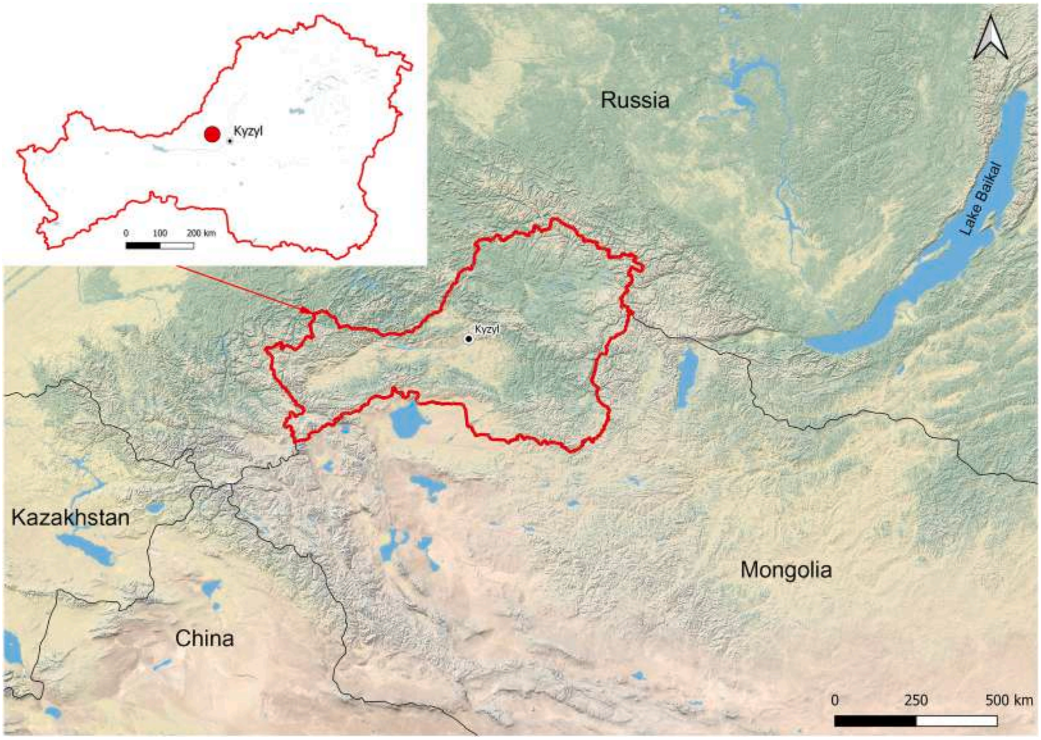

Fig. 1. Map of the Republic of Tuva (Russian Federation) showing the location of the Katylyg 5 settlement (red circle).

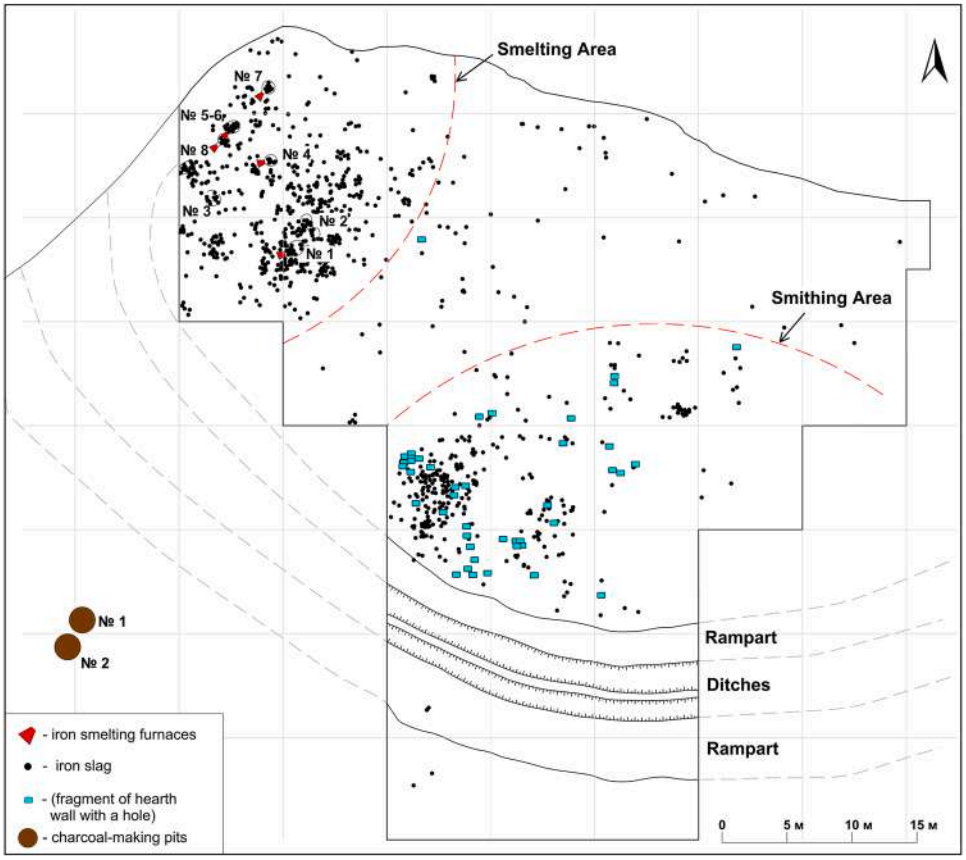

Fig. 2. The plan of the Katylyg 5 fortified settlement and the remains of the iron-smelting production.

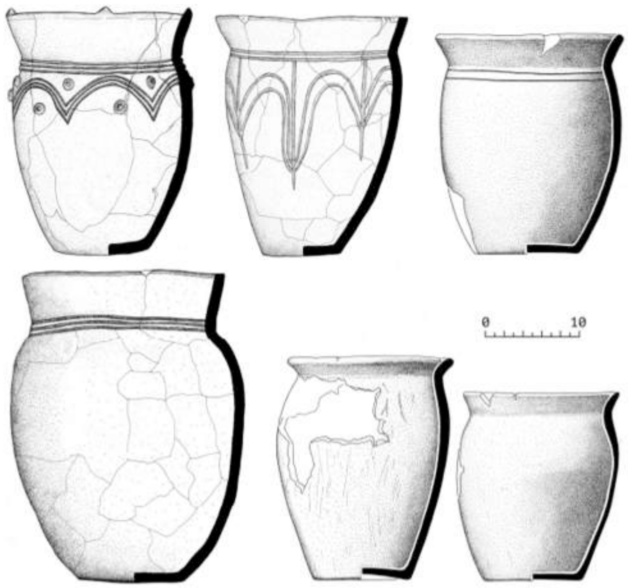

Fig. 3. Characteristic pottery from Katylyg 5.

Fig. 4. Slag blocks from smelting furnaces. A – Furnace No 1; B – Furnace No 4; C-D – Furnace No 6.

smelting was over prior to the chamber filling with slag.

No ore fragments were found at the site so far. However, small pieces could have been easily overlooked during excavations and may be found in the future.

An interesting feature of each furnace is the inclined underground channel, connecting the smelting chamber with the underground pit (Fig. 5). The fact that the channel was not filled with tapped slag indicates the existence of a barrier wall keeping the slag inside the chamber. Furthermore, as noted above, the slag-tapping occurred at the upper level. The possible function of the underground channel, therefore, remains to be understood. The scenario in which the channel was used to supply the air into the furnace is not supported by any evidence, although should not be discarded, especially, given the fact that the funnel-shape of the furnace could have created special air-dynamic conditions. Alternatively, the underground channel could have been used to monitor the slag formation and the overall progress of the smelt.

The fragments of clay tuyeres were also found at the settlement suggesting the use of bellows for the air supply. However, tuyeres were found not in situ, and the exact mechanism of the air supply system remains to be fully understood.

The total mass of the iron slag from the smelting zone: 1050 kg, including 990 kg of slag blocks from nine excavated furnaces and 61 kg of slag found around the furnaces. Based on this data, one smelt produced an average of 100 kg of slag. According to known experimental data, the weight of the produced slag typically constitutes 30–50% of the total mass of the smelted ore (Serneels and Crew, 1997; Senn et al., 2010). Therefore, the typical amount of roasted ore consumed per single smelt in the Katylyg furnace was ca. 320 kg, and all nine furnaces consumed ca. 2800 kg of ore. Ethnographic records from native people of Western and Eastern Siberia show that the typical metal yield from a traditional bloomery furnace is 8–20% of the ore weight (Vodyasov, 2018: 176). This type is represented by ground cylindrical constructions made of clay. In different cultures their size varies. In the 18th century, the Kondoma Tatars built small clay furnaces of 30 cm in height with the diameter of the base equal to only 15 cm.

Yakut furnaces were much bigger than the Tatar ones, and thus wrought iron blooms produced by the Yakuts was heavier. Waclaw Seroshevskiy described the Yakut furnaces as cylindrical constructions 110 cm high, with a top hole for filling coal and ore of around 30 cm in diameter and with a bottom hole for bellows.

With regard to the Yakut iron, Waclaw Seroshevskiy wrote in 19th century that 1.4–1.6 kg of iron could be made out of 16 kg of iron ore. The weight of Yakut wrought bloom ranged from 10 to 16 kg, and this iron was porous and covered with a layer of slag, and so it had to be repeatedly heated to be cleaned, as a result of which half of its weight was gone. Out of 16 kg of wrought iron maximum 10 kg of iron was produced (Seroshevskiy, 1993: 368). Strelov, however, provides some different data on the Yakut metallurgy. According to him, on average, out of 16 kg of ground ore 7.4–8.2 kg of iron was produced, that is, 50–57%. Another description of iron smelting process as practiced by the Yakuts indicates that from an equal amount of ore and coal (24.5 kg each), 8.2 kg of wrought iron was produced, that is, 33% of the total amount of ore (Strelov, 1928: 55-57). Gmelin writing on the Kuznetsk Tatars of Western Siberia reported an even greater amount of iron yielded – at 65% (Vodyasov, 2016).

However, if to consider significant losses of wrought iron after forging referred to neither by Strelov nor by Gmelin, we can see that the yield of iron varies from 8 to 20% depending on the quality of ore and technologies applied.

If this is extrapolated to the ancient iron production at Katylyg, taking into account that only half of the site was excavated, the average total yield from nine furnaces would be ca. 224–560 kg of iron metal, which is a rather high output.

2.2. *Smithing and charcoal making zones*

The south-eastern zone of the site was used for iron smithing operations (Fig. 2), as outlined by the finds of broken smithing cake slag (avg. diameter 8 cm) and square clay blocks, possible fragments of smithing hearths wall, having a 2.5 cm hole for the tuyere (Fig. 6A). In total, 50 fragments of such clay blocks were found at the site. The total mass of smithing cakes found in the smithing zone is 9.4 kg. However, since only half of the settlement was excavated, the obtained data should be regarded as minimally representative of the full scale of metal production there.

An area located 40 m to the south-west from the smelting zone (Fig. 2) was probably used for the production of charcoal, as deduced from the presence of two large pits with charcoal sediment and fired soil (Fig. 7). Pit No 1 has a diameter of 2.8 m, a depth of 0.8 m (Fig. 7A); pit

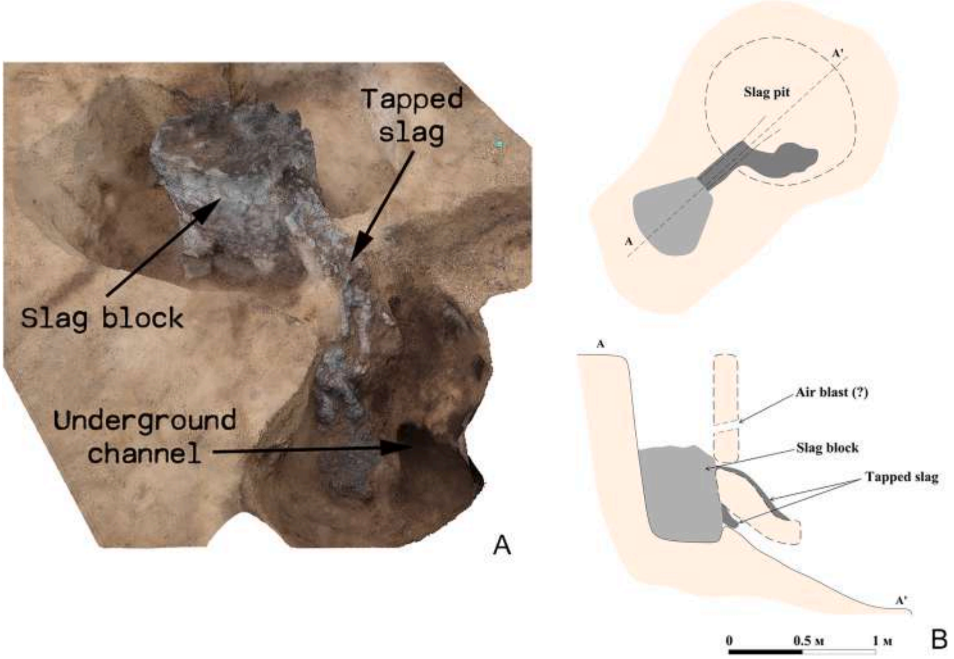

Fig. 5. 3D model (A) and schematic reconstruction (B) of the furnace No 5.

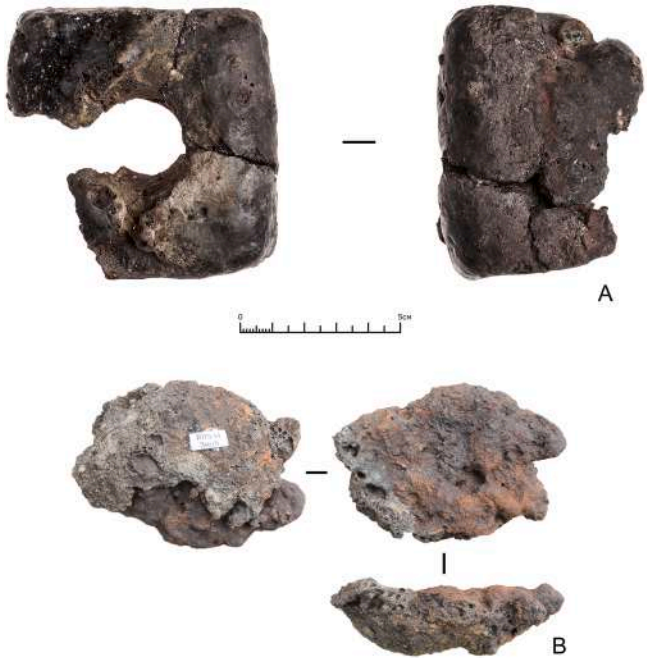

Fig. 6. The evidence of smithing activities. The walls of the smithing hearths (A); plano-convex smithing slag (B).

Fig. 7. Charcoal-production pits. A – charcoal pit No 1, B – charcoal pit No 2.

No 2 has a diameter of 2.8 m and a depth of 1.3 m (Fig. 7B). The bottom of pit No 2 was lined with stone slabs and covered with a thin charcoal layer. This evidence was not previously reported from Tuva and suggests that charcoal was produced by the simple combustion of wood with limited access to oxygen.

3. Chronology of Katylyg 5

Eight samples of charcoal from different zones of the Katylyg settlement were dated by the conventional radiocarbon analysis (Table 1). The calibration of dates was performed in OxCal 4.3 using calibration curve IntCal 20 (Reimer et al., 2020). The obtained dates indicate that metallurgical activities were performed sometime during the 3rd–4th centuries AD (Fig. 8), which is further supported by the finds of the Kokel pottery in the same stratigraphic layers with the smelting furnaces.

4. Methods

The methods of materials science analyses, applied to the slags and

Table 1
Radiocarbon dates from the Katylyg 5 fortified settlement. All dates were calibrated using OxCal v4.2.4 (Bronk Ramsey et al., 2013) and the IntCal20 calibration curve (Reimer et al., 2020).

Lab. Code	Archaeological context	Material	Dating technique	^{14}C Age (BP)	Cal AD (68.2%)	Cal AD (95.4%)
Le-10945	Trench z10, smithing area	Charcoal	conventional	1820 ± 45	133–325	88–349
Le-10943	Trench e13, smithing area	Charcoal	conventional	1770 ± 30	242–333	223–375
Le-10944	furnace No 5, smelting area	Charcoal	conventional	1770 ± 30	242–333	223–375
Le-10938	furnace No 6, smelting area	Charcoal	conventional	1760 ± 20	248–335	238–352
Le-10937	Trench e12, smithing area	Charcoal	conventional	1730 ± 20	256–375	250–404
Le-10940	furnace No 8, smelting area	Charcoal	conventional	1720 ± 20	259–380	254–406
Le-10942	Trench L12	Charcoal	conventional	1710 ± 30	262–402	252–416
Le-10941	Trench e11, smithing area	Charcoal	conventional	1700 ± 25	265–405	257–415

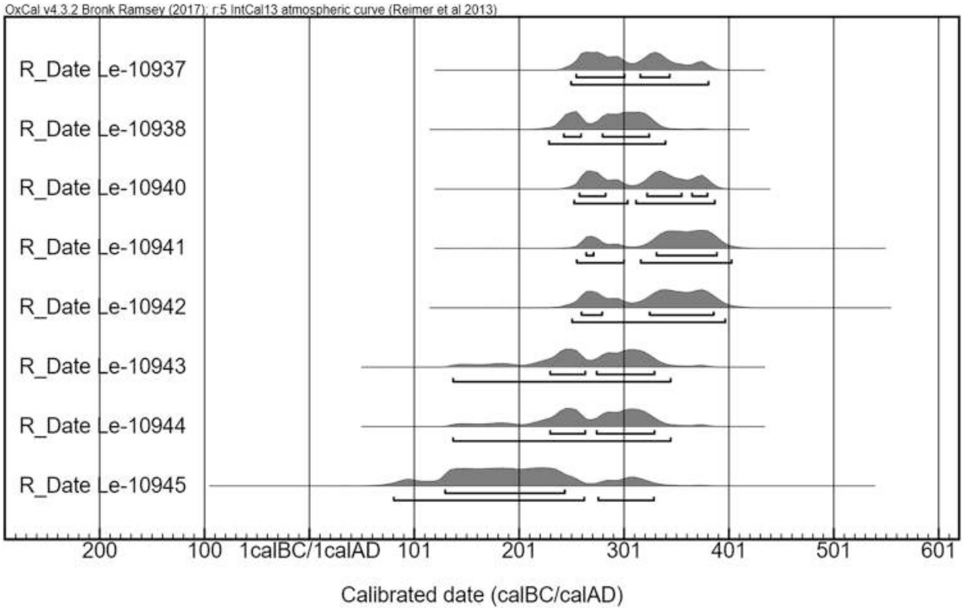

Fig. 8. Results of radiocarbon dating of the Katylyg 5 fortified settlement.

other metalworking products and residues, became commonplace for the understanding of various aspects about the past metallurgical technologies. In the studies of ancient iron production, the materials analyses can help to identify the nature (e.g., smelting, smithing), the RedOx atmosphere and the efficiency of technological processes, as well as the characteristics of the used raw materials (Selskienė, 2007, Charlton et al., 2010, Mei and Rehren, 2005).

For our study, we analysed a small assemblage (15 samples) of slag from Katylyg 5 including smelting (10) and smithing (5) slags. The full list of studied samples is provided in Table 2. We chose to focus our investigation on the slag because it is the most abundant type of metalworking remains at the site, which also preserves fundamental information about the technological process and the used raw materials. The choice of slag samples for analyses was based on their archaeological context. As this is a pilot study aiming to broader characterize the iron metallurgy at Katylyg 5, we sampled the slags from a range of smelting furnaces and trenches of the settlement. The specific goals were: (1) exploring the nature of technological processes that underlay formation of the slag; (2) verifying the identification of the slags from different production zones as formed during bloomery smelting and smithing processes; (3) identifying the mineralogical and geochemical markers of the smelted ores.

The slag samples were prepared as polished sections following the standard metallographic procedure and investigated using Optical Microscopy (N of analysed samples = 15), SEM-EDS (N = 14) and ICP-MS (N = 6). The Optical microscopy and SEM-EDS analyses were performed at the Institute of Mineralogy SU FRC MG UB RAS at the optical polarized microscope Olympus BX51 and SEM-EDS Tescan Vega 3 SBU equipped with EDA Oxford Instruments X-act.

SEM-EDS was used for analyses of individual phases/inclusions, performed in 14 slag samples, and 3–4 analyses of full areas (0.9 mm2), performed in 11 slag samples. The analyses were undertaken at following settings: accelerating voltage 20 kV, working distance to sample 15 mm, absorbed current at the cobalt reference – 260 pA, counting time for a peak –120 s; dead time – 10–15%. For control of the quality of analysis, we systematically analysed a cobalt reference standard. The operator at the SEM-EDS: I.A. Blinov. Full analyses of slags are provided in Supplementary S1.

To obtain the bulk trace element composition of samples, approximately 40–100 g of sample material was ground into a powder and analysed using ICP-MS. The ICP-MS analyses were performed at the Tomsk State University using Agilent 7500cx. The protocol for ICP-MS analyses is provided in Supplementary S2. The operator at the ICP-MS: E.S. Rabtsevich. The full analyses of ICP-MS are provided in Supplementary S3.

5. Results

5.1. Investigation of smelting slag

The morphological and microstructural features of Katylyg slag are summarized in Table 2 and shown in Figs. 9–10. The chemical analyses of slag by SEM-EDS and ICP-MS are summarized in Tables 3–5.

As the smelting slag from Katylyg appears both as furnace (i.e., solidified inside the furnace) and tapped slag (solidified outside of the furnace), both of these types were sampled and analysed.

Table 2
List of studied samples of Katilig slag and their main microstructural features.

#	Type		Sample No	Area	Microstructural features
1	Smelting slag	furnace	6/1	furnace 6	Contain crystals of hercynite
2		tapped	5/111	furnace 6	–
3		tapped	5/1	furnace 5	Contains relics of magnetite ore with small (2 μm) crystals of hercynite.
4		furnace	5/2	furnace 5	Contains magnetite ore relics and iron sulphide prills
5		furnace	1/2	furnace 1	–
6		furnace	8/1	furnace # 8	–
7		tapped	2/1	furnace 2	–
8		tapped	1/1	furnace 1	–
9		tapped/ furnace	N6/71	Trench n6, № 71	Contains iron prills rich in As
10	Smelting slag with semi-reacted ore		L6/6	Trench l6, № 6	Contains large portion of magnetite ore relicts and a crust of micro-crystalline quartz. Glass entraps prills (<1 μm) of iron sulphide
11	Smithing slag		zh12/ 22	Trench zh12	Contains Cu prills Metal Fe is only present as rims around wustite
12			e9/27	Trench e9, № 27	Contains corroded Fe (ca. 0.1–0.3% C). Contains rare inclusions (10–30 μm) of metal Cu
13			e9/40	Trench e9, № 40	Contains corroded Fe (0.2% and 0.6% C)
14			zh10/ 99	Trench z10	Contains corroded steel (≥0.8 %C)
15			zh10/6	Trench z10, № 6	Contains corroded Fe (0.3% C)

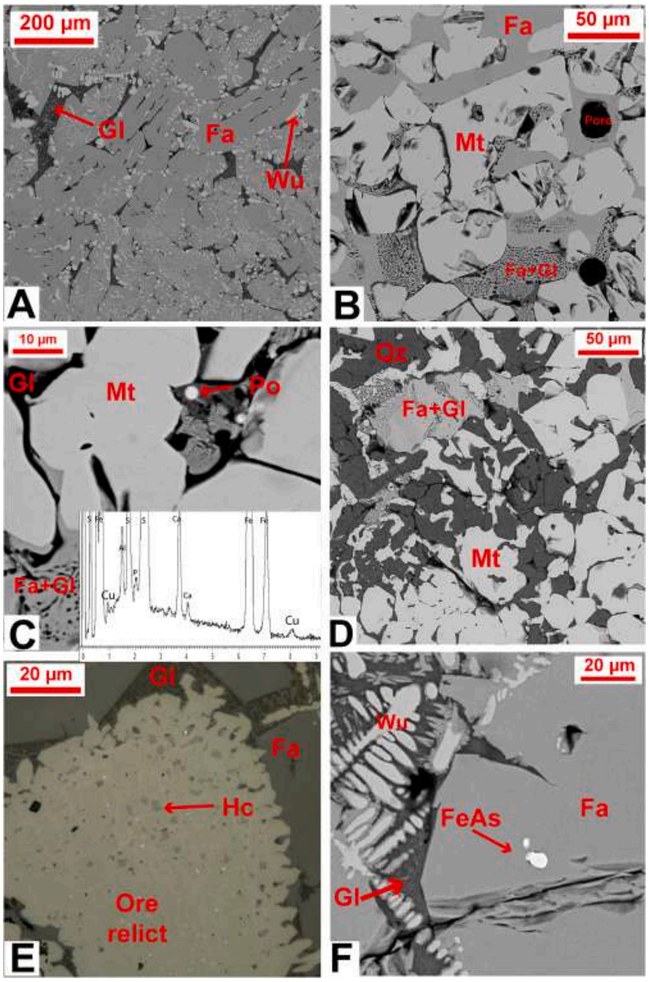

Fig. 9. The microstructural features of Katylyg smelting slag. **A.** 6-1. SEM. BEI. Overview image of the slag microstructure consisting of fayalite (Fa) laths, wustite (Wu) dendrites and glass (Gl) **B.** L6-6. SEM. Magnetite (Mt) ore relics in the matrix of fayalite and glass. **C.** L6-6. SEM. Pyrrhotite (Po) prills trapped in the glass and EDS-spectrum of pyrrhotite. **D.** L6-6. Area of the sample with large amount of microcrystalline quartz (Qtz). **E.** 5/1. OM. Grain of magnetite ore relict with inclusions of hercynite (Hc). **F.** N6-71. SEM. Structure with prills of iron arsenide.

Morphologically, the smelting slags are relatively dense, low-porous and have grey colour in section. Their microstructure reveals equiaxed or elongated crystals of fayalite, dendrites of wustite and a glassy matrix (Fig. 9A). The fayalite constitutes on average 75% of the slag, while wustite and glass comprise smaller portion; each up to 10–15%.

The bulk analyses confirm the identification of a typical bloomery iron slag (cf. slags from Switzerland, Serneels, 1993: Annexe 3–7), as it consists of (wt%) FeO (55–68), SiO2 (25–31), as well as smaller amounts of Al2O3 (3–6), and CaO (1–2) as the main slag-forming components (Table 3). However, two slags (no. 5/1 and 5/2) produced in the furnace 5 appear to be slightly more enriched in CaO (4.4–5.0) than the rest of the slags, which can be linked to the differences in the used raw materials or the greater contribution of the charcoal/clay.

The slag (no. l6/6) contains a large amount of partly reacted magnetite ore, identified by large (40–70 μm) rhomboid crystals (Fig. 9B). Apart from magnetite, the sample also contains fayalite intergrown with glass (Fig. 9C) and microcrystalline quartz (15–20 μm) (Fig. 9D). The glass from slag no. L6/6 is rich in SiO_2 (74 wt%) and also locally entrapped prills of neogenic iron sulphide (pyrrhotite or troilite) that probably formed by recrystallization of the precursor iron sulphide (Fig. 9C). The sulphide occurring in the slag contains admixtures of copper (ca. < 0.3 wt%) as seen from their EDS spectrum (Fig. 9D). This suggests that the original iron ores were associated with quartz and contained accessory amounts of iron sulphide minerals.

Inclusions of incompletely reacted magnetite ore (Fig. 9E) and pyrrhotite prills were also found in slags from furnace 5 (No 5/1, 5/2). Under high magnification, these ore relics reveal inclusions of metallic iron and crystals of hercynite. The latter incorporates small admixtures of TiO_2 (2 wt%) and V_2O_3 (0.6 wt%). A spinel crystal of similar composition ($TiO_2 = 2.5$ wt%, $V_2O_3 = 0.3$ wt%) is also found in slag no. 6/1 suggesting that the smelted ores contained impurities of Ti and V (Table 3).

In all smelting slags, small (<5–10 μm) inclusions of metallic iron also occurred. In two out of six samples (Nos 5/2; 6/1), metallic iron does not contain any elements detectable by the EDS analyses (Table 4), which is common for the typical composition of the bloomery iron. However, in three other samples (Nos 1/2, 5/111, 8/1), metallic iron contains impurities (wt%) of Co (0.5–0.6). Furthermore, the sample no. N6/71 contains metallic iron prills (Fig. 9F) of very unusual composition, close to iron arsenide (As levels: 0.8; 10.7; 34 wt%) also containing impurities of Ni (0.7–6.7) and Co (0.6–0.8). Overall, this data suggests that the used iron ores most likely contained some small amounts of Co, and occasionally Ni and As.

5.2. *Investigation of smithing slag*

The iron smithing slags from Katylyg 5 have characteristic morphological markers of secondary (forging) operations (Serneels and Perret, 2003; Dunster, Dungworth, 2012, Stepanov et al., 2020). These include: a plano-convex shaped cake, a rough top surface and a clay

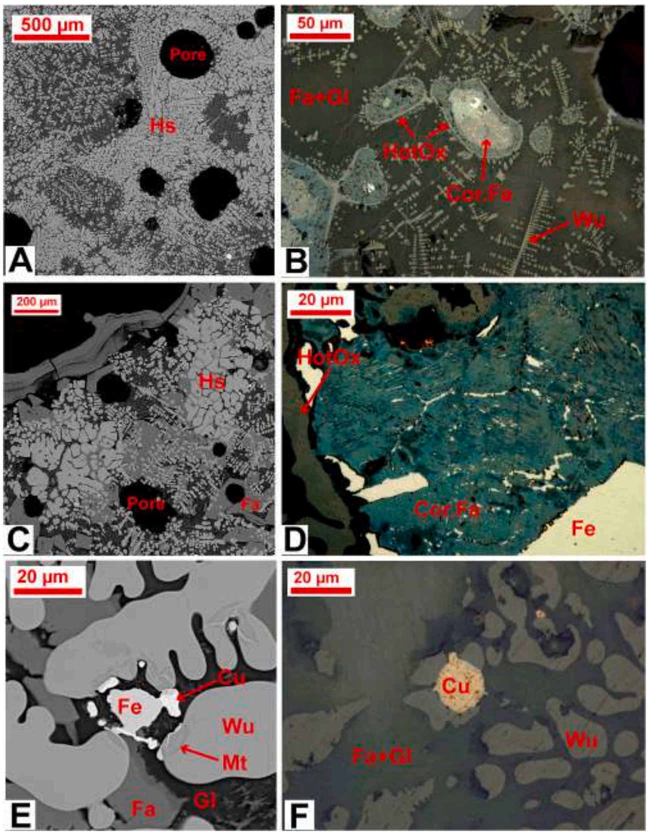

Fig. 10. Microstructures of smithing slag from Katylyg 5. **A.** zh12/22. SEM. Massive hammerscales (Hs) in the slag **B.** e9/27. OM. Hot oxidation (HotOx) rims around corroded iron in the matrix of wustite, fayalite and glass. **C.** zh10/6. SEM. Hammerscale agglomerates in the slag matrix. **D.** zh10/99. OM. Remnant carburized structures (Cor.Fe) 0.8% C contoured by hot oxidation rims. **E.** zh10/99. SEM. Iron (Fe) and copper (Cu) prills in the matrix of fayalite, wustite/magnetite and glass. **F.** zh12/22. OM. Copper (Cu) prill.

crust at the bottom surface (Fig. 6B). Upon sectioning, these slags reveal layered structures often marked by alignments of pores, and the presence of corroded metallic iron.

The OM investigation confirms that the slag was formed during hot oxidation of metallic iron, as it contains so-called "hammerscales" (Dungworth and Wilkes, 2007) often appearing as agglomerations of globular wustite ex-solved with magnetite (Fig. 10A, E) or as wustite/magnetite rims (Fig. 10B, D) around corroded metallic iron. This identification of morphological and microstructures markers of the smithing slags is also supported by experimental data (Brauns et al., 2020).

Mineralogically, the smithing slag mostly consists of the same phases (olivine, wustite and glass) that were found in the smelting slag, which is further supported by the similar bulk (full area) compositional analyses (Table 3). This suggests that the smithing slag was formed due to contribution from the hot oxidized metal and the smelting slag originally present in the pores of the forged metal.

The Katylyg smithing slags also contain remnant carburized structures, that is corroded pseudomorphs of the original metal structure, such as those identified in the slag from Southeastern Arabia (Stepanov et al., 2017, 2019). According to these remnant structures, the carbon content of iron from the smithing slag varies from one sample to another. In slag zh10/99, the forged metal was apparently hard steel based on the presence of remnant structures of 0.5–0.8% (Fig. 10D) and 1.5–2.0% C. In sample e9/40, the metal was probably heterogeneously carburized varying between mild (0.2 %C) and medium steel (0.6 %C), while in samples e9/27 and zh10/6, the metal could have been mild steel (0.2–0.3 %C). This suggests that various types of iron and steel may have been forged at the site. However, all of these alloy types could have been simultaneously present within a single bloom, which is often a very heterogeneous product (Pleiner, 2000). Only extensive analysis of a large number of various bloomery products and by-products from Katylyg can allow to characterize the nature of metal produced at the workshop.

One of the most striking features of the Katylyg assemblage of smithing slag is that four out of five Katylyg slags contain prills of copper-base alloys, often alloyed with metallic iron (Fig. 10E, F), similarly to some Iron Age slags from the Levant and Southern Caucasus (Erb-Satullo et al., 2020, Eliyahu-Behar et al., 2012). The copper-alloy droplets often grow at the periphery of metallic iron islands indicating the non-mixing between the two metals. The composition of the copper

Table 3
The chemical analyses (wt%) of smelting (smlt) and smithing (smth) slags by SEM-EDS, including bulk areas 0.9 mm^2 and individual analyses of some common phases (For full analyses, see Supplementary S1).

sample No	type	mineral type	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	Cl	K2O	CaO	TiO2	V2O3	FeO	BaO	Tot
L6/6	smlt	ore relict			0.7	1.1								98.2		100.0
		glass	1.18		4.03	74.1		0.37		1.48	1.3			17.1		99.5
		full area (n = 3)	0.4		1.1	16.6		0.3		0.1	0.3			81.3		100.0
1/1	smlt	wustite			0.5	0.9						0.6		98.0		100.0
		fayalite		0.3		29.8					1.0			68.3		99.5
		glass	4.8		21.2	41.5	0.7	0.9		5.0	8.5	0.2		17.6		100.4
1/1	smlt	full area (n = 3)	0.8	0.5	3.6	24.5				0.6	1.7	0.2		68.2		100.0
5/1	smlt	ore relict			1.1	0.8					0.2	0.3		97.6		99.9
		wustite				0.9								98.4		99.3
		fayalite		0.7		30.0					1.3			67.6		99.6
		glass	2.7		14.7	45.0	0.6	0.5	2.0	3.8	4.2	0.1		21.6		95.2
		full area (n = 3)	1.2	0.7	5.5	29.6				0.9	4.4	0.2		57.5		100.0
5/2	smlt	hercynite from ore relic			39.6	0.8					0.1	2.0	0.6	56.2		99.3
	smlt	full area (n = 3)	1.4	0.8	6.1	30.8				1.2	5.0	0.2		54.5		100.0
6/1	smlt	full area (n = 3)	0.8	0.6	3.0	25.4				0.4	1.0	0.1		68.8		100.0
N6/71	smlt	full area (n = 3)	1.1	0.6	5.7	28.1	0.2	0.2		0.9	1.9	0.3		61.2		100.0
E9/40	smth	full area (n = 4)	1.2	1.2	6.1	24.6	0.2			1.2	5.1	0.1		60.4		100.0
E9/27	smth	full area (n = 3)	1.3	0.9	5.7	22.9	0.1			1.1	5.1	0.2		63.1		100.2
Zh10/6	smth	full area (n = 3)	2.0	0.8	7.1	27.0	0.1			1.4	4.7	0.2		56.6	0.2	100.0
Zh10/99	smth	full area (n = 4)	1.4	1.2	5.8	24.5	0.7			1.4	5.6	0.1		59.2		100.0
Zh12/22	smth	wustite		0.5	0.8	0.4								98.8		100.4
		fayalite		3.0	0.4	32.2					9.2			55.0		99.7
		glass	3.5		17.5	40.8	1.7			5.5	11.1	0.4		19.4		99.8
		full area (n = 3)	0.9	0.9	4.1	17.6	0.3			1.1	5.8	0.1		69.2		100.0

Table 4
Composition of metal inclusions (wt%) from smelting and smithing slag of Katylyg 5.

Type	Sample No	N of analyses	Fe	Co	Ni	Cu	As	Total
smelting slag	1/2	n = 5	99	0.5				99.6
	5/111	n = 4	98.7	0.6	0.3			99.7
	5/2	n = 1	100.1					100.2
	N6/71	n = 3	97.0	0.8	0.7		0.9	99.7
		n = 2	86.6	0.8	2.4		10.7	100.5
		n = 1	58.7	0.6	6.7		34.1	100.2
	6/1	n = 1	100.7					100.7
	8/1	n = 4	99.6	0.5				99.6
smithing slag	e9/40	n = 2	98.7			0.7		99.6
	e9/27	n = 1	2.7			97.2		99.9
		n = 1	7.9			91.8		99.7
		n = 3	98.1			1.8		99.9
	zh10/6	n = 1	100.3					100.3
	zh10/99	n = 4	6.6			93.7		100.3
		n = 1	5.5		0.3	94.2		100.0
		n = 1	94.2	0.7	0.5	5.3		100.7
		n = 2	92.0	0.5		7.4		99.9
	zh12/22	n = 2	5.2			94.2	0.7	100.1
		n = 2	3.7		0.6	94.9	1.6	100.8
		n = 1	99.5					99.7

Table 5
Analyses of some trace elements from smelting and smithing slags of Katylyg 5 by ICP-MS (full trace elemental data is provided in Supplementary S2).

Sample No	Type	Li	Ti	V	Cr	Mn	Co	Ni	Cu	Zn	Sr	Mo	Ba	Pb
1/1	smlt	4	1049	136	21	459	25	6	14	20	78	4	179	1.5
5/1	smlt	4	1213	115	193	480	32	46	18	32	138	8	207	2.0
N6/71	smlt	6	1416	144	24	427	24	7	12	19	93	1	183	1.3
e9/40	smth	11	1153	46	46	466	24	18	988	8	150	13	248	0.7
zh10/6	smth	10	1264	32	41	585	37	28	153	12	220	2	365	1.1
e9/27	smth	8	1065	44	38	546	35	26	1322	7	145	14	248	1.1

prills incorporates iron (3–8 wt%), nickel (avg. 0.5 wt%), and arsenic (up to 1.6 wt%; only in slag zh12/22). Likewise, the metallic iron contains minor levels of copper (0.7–7.0 wt%), and in case of slag zh10/99, impurities of cobalt (avg. 0.6 wt%) and nickel (avg. 0.4 wt%). Overall, these features suggest that bronzes were worked in the same hearths where iron was forged.

5.3. Comparison of chemical composition of smelting and smithing slag

Despite the apparent compositional similarities between smelting and smithing slags, some minor compositional differences are also attested. In the smithing slag, the wustite, fayalite and glass are often characterized by slightly higher MgO and CaO contents than in most of the smelting slag (Table 3). These differences in contents of MgO and CanO, as well as other alkaline/alkaline-earth elements (Na_2O, K_2O, Li, Sr, Ba) are reflected in the bulk composition of the smelting and smithing slag (Figs. 11, 12, Table 5). In contrast to most of the smelting slags, the elevated contents of CaO, K_2O, MgO in the smithing slags result in higher ratios of these elements with SiO_2 and Al_2O_3 (Fig. 11). However, unlikely most of the smelting slags, the slags no. 5/1, 5/1 (encircled in Fig. 11) due to their higher CaO contents group together with the smithing slags on bivariate plots Ca/Al, Ca/Si and Ca/K.

Overall, the higher contents of alkaline/alkaline-earth elements (Na_2O, K_2O, Li, Sr, Ba) in the smithing slag may be due to extra contribution of the charcoal (Crew, 2000, Charlton et al., 2010: Table. 1). The formation of smithing slag can be imagined as all the smelting slag entrapped in the forged bloom/iron + hot oxidized metal + ash from the charcoal used during smithing, which therefore would explain the higher total contribution of charcoal ash into the smithing slag than in the smelting slag.

The higher ash input during the smithing process therefore could have partly concealed the original ore signature. Apart from alkaline elements, the contents of alumina and silica of the smithing slag can be also affected by the melted hearth lining or the use of mineral fluxes (e.g., Workman et al., 2021, Serneels and Perret, 2003), which could have also happened during formation of Katylyg smithing slag.

Alternatively, the variation in ratios Ca/Si, Cal/Al, Ca/K, Si/Al among the slags from Katylyg could have been caused by the ore composition. For instance, different procedures of ore preparation, particularly non-complete removal of the calcium carbonate gangue from the ore prior roasting could have ultimately resulted in higher bulk CaO contents of the smelted ore and the deriving slag. As discussed in the Section 6.1, magnetite hosted by calcic skarns was one of the likely ore sources smelted at Katylyg 5, and the use of these ores, without significant removal of their gangue, can be potentially reflected in higher CaO contents of some smelting slags (e.g., 5/1 and 5/2).

The bivariate plots also show that slag no. L6/6 incorporating high amount of relicts of semi-reacted magnetite ore is characterized by similar to the rest of the smelting slags ratios of Ca, Si, Al, K and Mg (Fig. 11). This suggests the likelihood of smelting of quartz-magnetite ore at the settlement.

The smelting slag also has higher V contents (avg. 130 ppm) than the smithing slag (avg. 40 ppm) (Fig. 12, Table 5). Given the fact that vanadium was found in spinels from ore relict grains, this element must be predominantly contributed by the ore. According to the Ellingham diagram (Craddock, 1995: 190), during bloomery smelting vanadium does not partition with the ore and instead almost fully passes from the ore into the slag. Therefore, during the iron smithing process, the bulk amount of vanadium is diluted in the forming smithing slag due to the large contribution from the hot-oxidized bloomery iron, which is mostly free from vanadium. Similar depletion effect from smelting to smithing slags was also reported for the contents of MnO by McDonnell (1986), and can be probably observed for the contents of Cr_2O_3, since Cr, Mn and V are reduced under similar oxygen partial pressures.

The implication of this data is that contents of V, Mn and Cr, if present in sufficient amounts in the ore (i.e., significantly surpassing

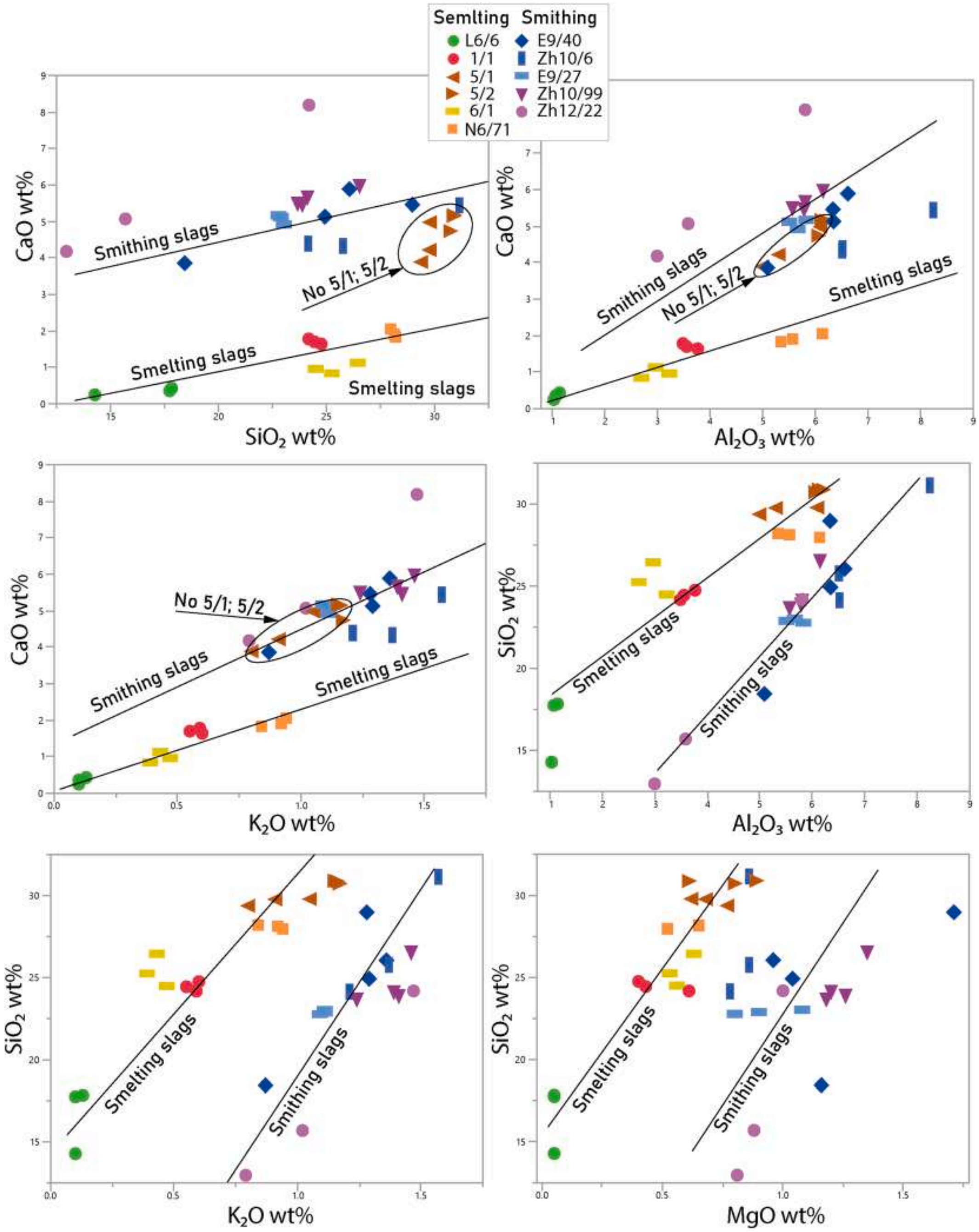

Fig. 11. Bivariate plots of contents of major elements (CaO, SiO_2, Al_2O_3, K_2O, MgO), based on the results of full-area SEM-EDS analyses, in the smelting (N = 6) and smithing (N = 5) slags from Katylyg 5.

contents of these elements in the clay to neglect the contamination effect) can be potentially used for chemical differentiation between smelting and smithing slags. However, this discrimination would only be possible if smelting and smithing slags are part of the same chaîne opératoire, in which chemically homogeneous ore is smelted. If the ore is heterogeneous, which was often the case of past metallurgical practices, or several different ore sources are used, or contents of Mn, V or Cr of the ore do not significantly surpass contents of these elements in the clay, then chemical differentiation between smelting and smithing slags would not be possible.

In accordance with microstructural observations (i.e., presence of copper prills), the bulk analyses of the smithing slag are characterized by higher levels of Cu, in contrast to the smelting slag. The contents of Cr, Ni, Co and Mo vary within the dataset of six slag analyses, possibly due to the variation of these elements in the composition of the smelted ores or in the composition of worked copper-alloys (which can be only reflected in the composition of the smithing slag).

Although a small number of REE analyses of slag does not allow to fully explore the range of local geochemical signatures, certain compositional trends can be nevertheless discerned (Fig. 13). Significantly, the distribution of rare earth elements (REE) and some other ore-signifying elements (Th, U, Nb, Hf) suggests that three smelting and smithing slags, except outlier no. 1/1, were produced from a similar ore. Furthermore, the analyses reveal the minor variation in Eu anomaly between slags no. e9/27, e9/40 (neutral Eu anomaly) and slags no. 1/1, 5/1, N6/71 (weakly negative Eu anomaly). This variation could have been caused by the natural heterogeneity within the ore deposit; by the incorporation by the slag of minerals such as plagioclase, which can

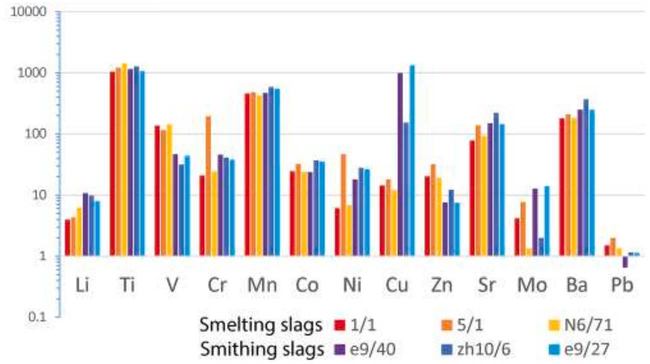

Fig. 12. Contents of trace elements in the smelting and smithing slags from Katylyg 5.

concentrate Eu; or by the exploitation of several ore sources. The latter hypothesis is also supported by the sharply outlying REE trends of slag no 1/1.

It is also worth emphasizing that smelting slag no. N6/71 containing iron arsenide prills does not outlay on the REE graph indicating a geochemical source similar to the rest of the slags.

Overall, the archaeometric investigation of slag from Katylyg, although bears only preliminary nature, provides key information about the metallurgical processes in which the slags were formed. Significantly, the analyses reveal that: (1) the slags derive from bloomery iron production; (2) two spatially separated areas of the site are smelting and smithing production zones; (3) the microstructures of most of the iron-smithing slags preserve traces of copper-working operations. Finally, the analyses allow the identification of possible mineralogical and geochemical markers of the smelted ore, as discussed below.

6. Discussion

6.1. The sources of iron ores

The distribution of REEs in Katylyg slag is similar to that of the skarns from the Co-Cu-As Khovu-Aksy deposit (Gusev, 2019: Fig. 3), located ca. 70 km to the south from Katylyg, (Fig. 14) suggesting a similar provenance. The calcic skarns from Khovu-Aksy ore field often host various mineralization including Au, Cu-As-Co, as well as Fe occurring as magnetite (Aleksandrovsky et al., 2008; Zaikov et al., 1981).

In the 50–100 km radius from Katylyg, magnetite and hematite are the main ore types (Fig. 13), which were intensively mined in the past (Alexandrovskiy et al., 2008: 158, Sunchugashev, 1969: 122, 133). Magnetite ores are often associated with calcic (carbonate) skarns and copper mineralization (malachite and azurite) (Sunchugashev, 1969, Fig. 49) and were probably also used at Katylyg, as deduced from the presence of magnetite relicts in three analysed smelting slags including slag no. L6/6 characterized by high portion of ore relicts. Alongside calcium carbonate, the quartz was probably another major mineral association of local magnetite ores as evidenced from abundant occurrence of quartz in slag no. L6/6. The common utilization of quartz-magnetite ores at the site is further supported by the similar ratios of Si/Al, Ca/Al, Ca/Si, Ca/K, Si/Mg in slag no. L6/6 and three smelting slags (1/1, 6/1, N6/71). Among other geochemical characteristics of the smelted ores that can be used in future provenance studies, are elevated vanadium contents, minor impurities of sulphur, and REE distribution characterized by weakly negative Eu anomaly and depletion of heavy REE compared to light REE.

Overall, the skarn-hosted magnetite deposits of Tuva, including calcic skarns from Khovu-Aksy ore field, are often associated with copper ore deposits, and therefore can incorporate trace amounts of chalcophile elements (Lebedev, 2012). In Katylyg slag, this is seen from the small admixtures of Cu (up to 0.3 wt%) in the neogenic iron sulphide (pyrrhotite) prills. Furthermore, the impurities of Co in the prills from smelting slag from Katylyg support the possibility of smelting of iron ores directly or indirectly associated with polymetallic ores of the Khovu-Aksy deposit.

A more direct indicator towards the use of ores from the Khovu-Aksy field is the composition of prills in sample N6/71. Apart from iron, these prills contain high amounts of As and Ni and can be classified as speiss. However, the deliberate use of speiss for production of iron seems unlikely, since the small amount of arsenic, as well sulphur often associated with it, would render the iron extremely brittle and unforgeable (Davis, 2001: 164). Therefore, the identified speiss prills are not necessarily representative of the whole bloom composition and may be present due to the heterogeneity of the smelted ore batch. Alternately, these prills reflect the experimentation attempts with non-common types of iron ores, such as gossans of polymetallic Cu-As-Co deposits.

Overall, the Khovu-Aksy polymetallic ores were apparently exploited for copper at least since the Iron Age, given the finds of characteristic cultural materials next to trace of past mining activities, and the chemical analyses of the Iron Age copper objects from Tuva (Sunchugashev, 1969: 39, Append. 1–3, Zaykov et al., 2016: 232). The

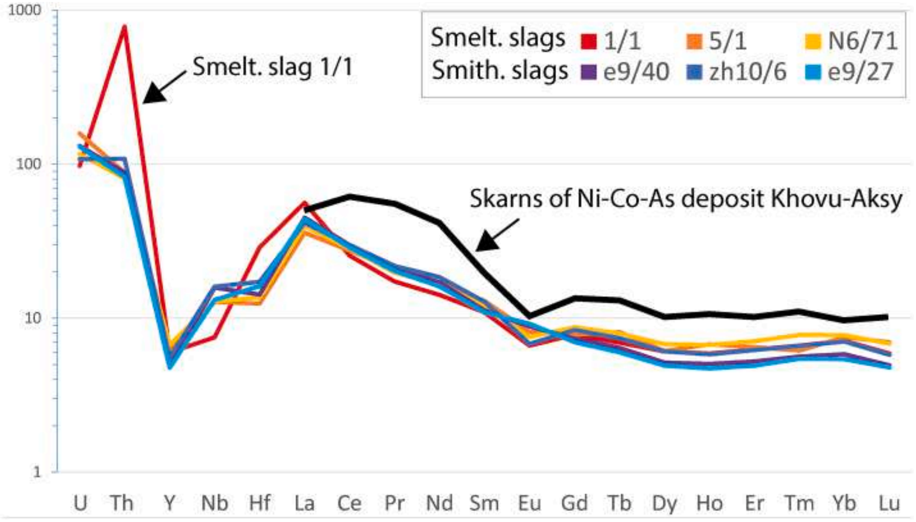

Fig. 13. Chondrite-normalized (McDonough and Sun, 1995) distribution of REEs and some other trace elements (U, Th, Y, Nb, Hf) from Katylyg slags. Graph also includes analysis of xenoliths of Cambrian metabasites from the skarns of Ni-Co-As ore cluster Khovu-Aksy (Gusev, 2019: Fig. 3).

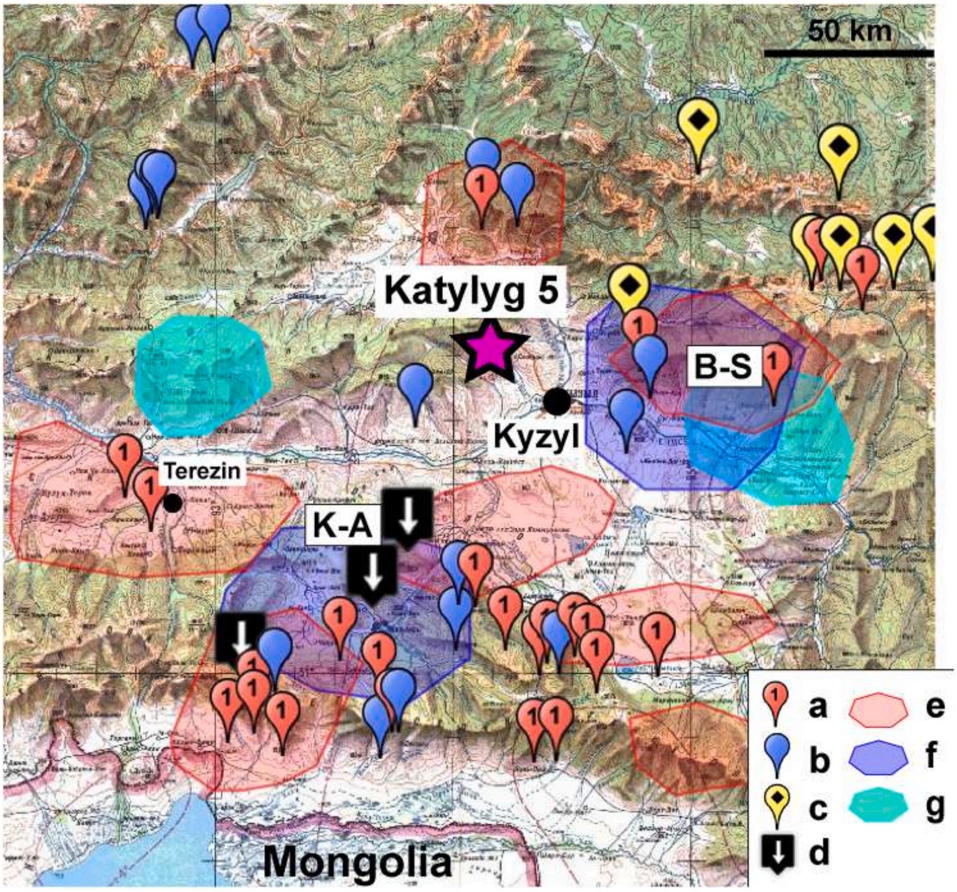

Fig. 14. Map of ore mining and smelting areas in the vicinity of Katylyg 5. **A.** Main iron ore deposits. **B.** Main copper ore deposits. **C.** Pb-Zn-Cu ore deposits. **D.** Co-Ni-Cu ore deposits. **E.** Ancient iron ore smelting and mining areas mines. **F.** Ancient copper ore smelting and mining areas. **G.** Gold placer deposits. Map compiled based on the data from Lebedev, 2012 (Fe deposits), Alexandrovskiy et al., 2008 (Pb-Zn-Cu deposits), Sunchugashev, 1969 (Cu and Fe past mines and smelting sites), Zaykov et al., 2016 (Au placer deposits). Abbreviations: K-A: Khovu-Asky ore field, B-S: Bay-Syut valley.

exploitation of these ores probably continued during subsequent Xiongnu period, as seen from the composition of copper objects from the Terezin cemetery, located ca. 70 km to the north-west from these ore deposits and ca. 100 km to the south-west from Katylyg (Leus, 2011). The Terezin objects contain high levels of As (5–20 wt%), minor amounts of Sb, Ni and trace levels of Co, Bi and Ag (Khavrin, 2011), which is generally consistent with the Khovu-Aksy geochemical signature. Finally, the use of these ores could have extended into the Xianbei period, as deduced from the presence of impurities of Co, Ni and As in the copper prills from at least two Katylg smithing slags (Zh12/22, Zh10/99). Given the potentially long exploitation history of Khovu-Aksy resources for copper, the possibility of their use in iron smelting at least since the Xiongnu time must also be considered in future studies.

Apart from the Khovu-Aksy field, numerous other deposits that contain traces of mining activities are known in Tuva. Several such sources are located ca. 40–65 km east (Bay-Syut valley) and north from Katylyg (Sunchugashev, 1969, Zaykov et al., 2016: 223), which is slightly closer than the Khovu-Aksy field (ca. 70 km).

It is also worth considering the possibility of the use of ores outcropping in the vicinity of Katylyg, given that the transportation of ca. 320 kg of ore needed for one smelt over dozens of kilometres would have been a laborious effort requiring well-developed logistics. Finally, it is also possible that more than one ore source was supplied to the settlement, as deduced from the variation in major element and REE composition of six analysed slags. Future studies aiming at the analyses of a larger assemblage of slags and ores from Katylyg will allow to develop a more detailed understating of the ore sources supplied to the site.

6.2. *Organization of iron production*

The undertaken study reveals the unique character of iron production in late Iron Age Siberia. For the first time we present clear evidence for the performance of iron smithing operations. This novelty is, of course, due to the low density of modern archaeometallurgical research in this region leaving no doubt that more iron smithing remains will be identified there in the future.

The presence of carburized structures in some corroded iron prills from the smithing slag, suggests that these smithing slags were formed by forging steely or heterogeneously carburized iron. Such iron could have been produced via secondary carburisation, which was already practised by the Xiongnu in Mongolia (Park et al., 2010); or via primary smelting in the bloomery furnace (e.g., Charlton et al., 2010). Both of these possibilities are speculative at present and can only be addressed by analysing a larger set of samples of slags and iron objects, with a particular focus on the investigation of heterogeneity of carbon distribution in the metal.

The character of the organization of iron smelting and smithing activities at Katylyg 5 is particularly distinctive. The area of iron smithing activities is still separated from the smelting zone by 15 m. Although located not far apart from one another, no finds of smithing slags were found in the smelting area and vice versa. Such disconnection between smelting and smithing activities may reflect the large-scale and/or high intensity of metallurgical operations at the site. Given the high amounts of ore and charcoal required for a single smelt in a Katylyg furnace, a high degree of preparation, organization and overall labour investments was probably needed to better control the process (cf. ethnographic records of African smelting, David et al., 1989). At Katylyg, this control was probably achieved by separating the working zones of the smelting and smithing, therefore allowing each of the specialist groups to better perform their work. The failure to control such process would have risked yielding a low-quality iron product, therefore, nullifying all invested labour resource (cf., the experiments by Leroy et al., 2020).

Despite the relative separation of the smelting and smithing zones at Katylyg, they are still quite close to each other and to the charcoal-making zone indicating that all metallurgical operations were performed in one place. Given the taiga forest location of Katylg (i.e., not the steppe zone!), the site could have been intentionally chosen not only due to the possible proximity to the ore sources but also due to the proximity to the forest needed for supplying metallurgical fuel.

The proximity between smelting and smithing zones was also reported for contemporary iron production centres of Cis-Baikal (as discussed in Section 6.3). This arrangement of Siberian iron workshops is different from many European or African centres where the smelting and smithing sites were often separated by many kilometres, as the smelting was performed near the ore deposits, while the smithing occurred in the settlements where there was a demand for finished iron objects (LaViolette, 2000, Bauvais and Fluzin, 2009).

The direct rationale for the spatial joining of smelting and smithing zones may conclude in the more effective refining of the freshly hot blooms, avoiding excessive losses of metal on its reheating. This connection between smelting and smithing products at Katylyg is also deduced from the similar major and trace element composition of smelting and smithing slag.

The more general benefit from the performance of all metallurgical operations at one site is that such chaîne opératoire probably allowed for a more self-sufficient subsistence of nomads not requiring them to rely on complex exchange networks. Another possibility is that the commodity produced at the site could have mostly been a semi-product (bar, ingot, billet or a bloom cut), that was traded and shaped into a finished item elsewhere.

Alternatively, such organization of labour could have been controlled more easily. The metalworkers could have been connected to the clients/patrons for whom the metal was produced and therefore directly responsible for assuring production of good quality iron. In order to consistently produce sufficient quantities of forgeable metal (i.e., not contaminated with harmful impurities as could be the case of bloom produced alongside slag n6/71), the process might have required supervision throughout all stages, from the ore section to the manufacture of the finished object.

6.3. Connection between iron and copper metallurgy

A striking observation made from the analysis of Katylyg slag is that the site's iron-making industry was apparently connected, both at the scale of mining and subsequent processing, to the industry of copper-making, similarly as it was in the Caucasus and possibly the Levant during the Iron Age (Erb-Satullo et al., 2020, Workman et al., 2020). The various local metallic resources including copper and iron ores were extracted, smelted (although evidence for copper smelting was not found yet) and worked at Katylyg during 3rd-4th c. AD.

The analysis of copper prills from iron smithing slag, although not representative of the full scale of bronze-working activities, suggests that at least some simple bronze-working operations such as heating, hot-forging, possibly melting of copper in crucibles and casting were performed by the Katylyg blacksmiths. The iron ores that were smelted at the site were geologically associated with copper ore deposits indicating that the miners familiar with one type of metallic resources were also able to locate the others. Despite this association, the exploited iron ores did not contain copper as it was not found in any of the smelting slag as prills, nor formed an alloy with the metallic iron. This fact suggests that the miners supplying ore to Katylyg apparently had sufficient skill to prospect good source of iron ores that were mostly free of harmful impurities such as Cu, S or As.

The natural richness of resources in Tuva, including Fe, Cu, Au, As-Cu, Pb-Cu ores, apparently also contributed to the important mining role of this region at least since the Final Bronze Age (c. 1300 BCE) and into the later periods.

It seems also very peculiar that despite the likely division of Katylyg metalworkers into smelters and blacksmiths, it was the blacksmiths who performed the bronze-working at the site. This fact allows us to raise the questions about the character of integration of iron and bronze-production economies, and the specialization of Eurasian metal craftsmen during early 1st millennium AD.

By the time of metalworking activities at Katylyg (3rd-4th c. AD), bronze had been fully substituted by iron in weaponry and tools and continued to be used only in decorative objects/jewellery and castings (Zavyalov and Terekhova, 2015, Sunchugashev, 1979: 168). Therefore, the demand for elaborate bronze products and the rationale for the development of specialized bronze industry probably did not outweigh the demand for the more essential iron-based economy, particularly in Tuva. Therefore, in many parts of the Southern Siberia, development of specialization in iron smelting and blacksmithing was probably more essential for subsistence than specialization in the bronze-making. This along with the socio-cultural context of the Katylyg settlement can explain the performance of copper-working by the blacksmiths and not by a separate class of bronze-smiths.

6.4. The chronology of trapezoid underground furnaces

The chronology and origins of the tradition of iron smelting in trapezoid underground furnaces with the inclined rear wall and an underground channel, used at Katylyg, represents one of the main questions of the study. To address it, we need to consider typological parallels with the other furnaces from Tuva and adjacent regions. Some of these parallels can be noticed in a furnace from the Bay-Syut valley, at the site of Turlug (Fig. 15A, section A-A'). The underground installation, along with another one connected to the same slag pit, was excavated by Sunchugashev (1969: Fig. 54) who also noted its poor preservation. According to Sunchugashev's scheme, the Turlug installation similarly to Katylyg furnaces had an inclined read wall. However, its top was not trapezoid, but rhomboid in plan.

Most other underground furnaces from the Bay-Syut valley show few similarities with the Katylyg installations as they have a rectangular or prismatic chamber with straight vertical walls (Fig. 15A, section B-B') and were often operated in pairs, which is not attested for at Katylyg.

Although Sunchugashev himself noticed that the typological variations of furnaces can potentially reflect their different chronologies, he proposed to date the Bay-Syut furnaces to the 3rd-2nd c. BC based on pottery finds from the identified ore mining area located 16–18 km away from the furnaces (Sunchugashev, 1969: 107-108). In our view, even if the pottery is indeed from this period, it dates the mines and not the smelting sites. In fact, the smelting furnaces dated to the 3rd-2nd c. BC were thus far not found in Tuva, although the presence of the 3rd c. BC pottery in mines is a strong argument for the existence of iron metallurgy in Tuva during the Xiongnu period (300 BCE – 100 CE).

Although the trapezoid in plan underground furnaces were not found in the adjacent regions of Khakassia, Altai and Mongolia, similar installations were discovered at archaeological sites from the western shore of Lake Baikal (Cis-Baikal, located 900 km from Katylyg 5): Barun-Khal 2, Barun-Khal 3, Kurminskoye Ozero 1 and Kurma 28 (Kharinsky and Snopkov, 2004; Kozhevnikov et al., 2018; Snopkov and Kharinsky, 2019). The Cis-Baikal installations, similarly to the Katylyg furnaces, have an inclined rear wall and an underground channel. In contrast to the Katylyg installations, the Baikal furnaces have also several typological distinctions (Fig. 16B) including slightly larger dimensions of the smelting chamber, a steeper inclination of the rear wall and a different spatial layout (see, Kozhevnikov et al., 2018: Figs. 10, 16).

The Cis-Baikal furnaces dated by the conventional method to the broad range 3rd c. BC – 10th c. AD. Given that the charcoal was often produced from larch trees (Snopkov et al., 2012) whose average age can reach up to several hundreds of years, it is possible that the oldest

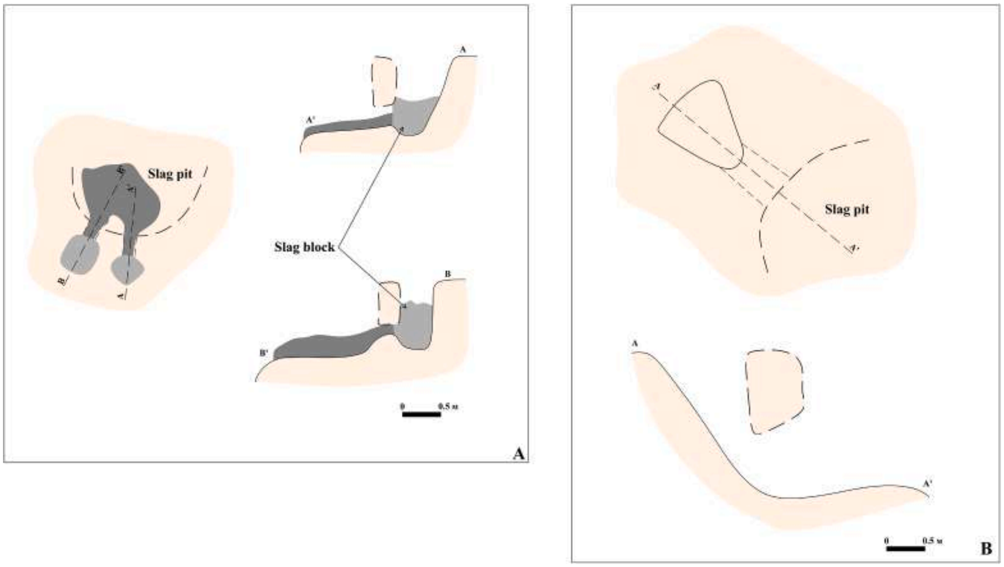

Fig. 15. Iron smelting furnaces from Tuva (A) and Cis-Baikal (B) that show slight or pronounced typological similarities with the Katylyg furnaces. Taken from (Sunchugashev, 1969: Fig. 54; Kharinsky and Snopkov, 2004: Fig. 7).

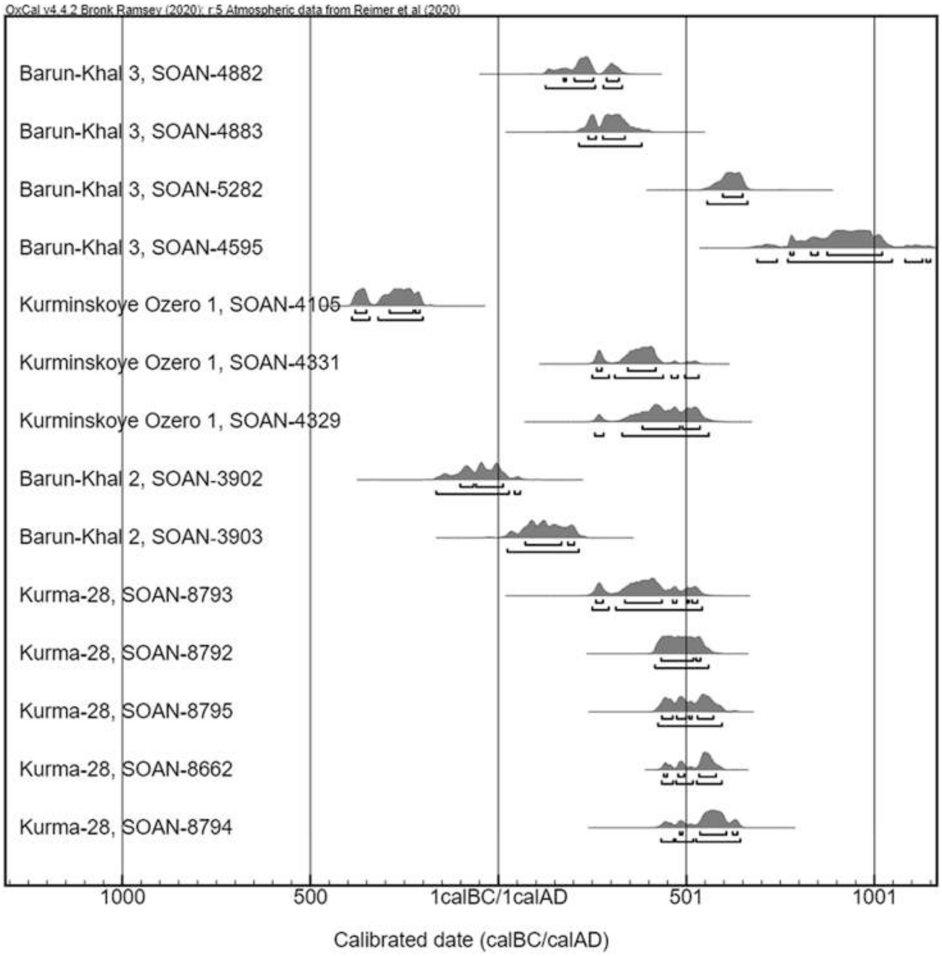

Fig. 16. Radiocarbon dating of charcoal from the trapezoid underground furnaces of Cis-Baikal. The data taken from (Kharinsky and Snopkov, 2004; Kharinsky et al., 2012; Kharinsky et al., 2013; Kozhevnikov et al.2018). All of the dates were calibrated using OxCal v4.2.4 (Bronk Ramsey et al., 2013) and the IntCal20 calibration curve (Reimer et al., 2020).

returned radiocarbon date (SOAN-4105, Fig. 14, Kharinsky and Snopkov, 2004) provided an older date than the actual time of smelting due to the "old wood" effect as shown at an example of iron smelting in Altai region (Vodyasov et al., 2020, Vodyasov and Zaitceva, 2020). The rest of the charcoal dates from the Baikal furnaces (Fig. 16), however, indicate that they emerged in the region at the end of the 1st millennium BC – early 1st millennium AD, continuing to be used over most of the 1st millennium AD. Disregarding the local typological variations between Katylyg and Baikal installations, the general similarities and the earlier dates of Baikal furnaces suggest that the technological tradition of trapezoid furnaces spread from the Baikal region into Tuva sometime during the first half of the 1st millennium AD.

6.5. The social context of early iron smelting in Southern Siberia

The archaeologists who excavated the Cis-Baikal furnaces associated them to the Elga culture (3rd c BC – 4th c. AD), based on radiocarbon dating of charcoal, associated finds of smooth-walled pottery and the cultural materials from nearby graves. The Elga culture is characterized by a specific burial tradition, as well as material culture finds including iron objects, smooth-walled pottery, open-work belt plates, spoon-shaped pendants and belt buckles – all thought to have been influenced by the Xiongnu culture (Kharinsky, 2001, Kharinsky, 2014).

Overall, in Southern Siberia and Mongolia, the Xiongnu period is characterized by the abundance of iron objects (Sunchugashev, 1969, 1979, Kharinsky, 2001, 2014, Brosseder and Miller, 2011, Houle and Broderick, 2011; Zavyalov and Terekhova, 2015) and by the emergence of iron-smelting remains (Vodyasov and Zaitceva, 2020).

The Xiongnu furnaces are often oval underground installations of relatively small size (height 0.5–0.8 m, width 0.3–0.6 m, length 0.6–1.1 m) and a moderate capacity (ca. 0.12–0.25 m3) (Sunchugashev, 1969, 1979, Sasada and Chunag, 2014). Although none of these furnaces can be clearly identified as trapezoid-type, these furnaces reveal general similarities with the trapezoid installations, including the arrangement of the furnace chamber in the dug clayey soil, the presence of underground channels, the similar dimensions and similar capacity.

The technological practices, including iron smelting, set and adopted by the Xiongnu rule probably continued past the disintegration of the Empire (1st-2nd centuries AD) as evidenced by the continuing existence of some Xiongnu-influenced cultures of South Siberia. At the same time, the new socio-cultural entities such as Kokel culture, to which inhabitants of the Katylyg 5 were identified, appeared after the breakdown of the Xiongnu at the early 1st millennium AD. In this sense, it is important to notice that the formation of the Kokel culture in Tuva can be explained by the westward waves of migration as a result of Xianbei expansion during 1st-3rd c. AD (Sadykov, 2017, 2018a). As a direct or indirect consequence of these events, the new tradition of iron smelting using trapezoid furnaces could have appeared in the Southern Siberia. This could have been an original and independent innovation brought from elsewhere since long-distance migrations were already common during the Xiongnu period (Jeong et al., 2020), or a modification of the previous local smelting tradition to better answer the demand created by the new political powers. Obviously, the richness of metallic resources of South Siberia was another major factor in these technological changes.

The new political powers probably stimulated the creation of the new economic networks, through which iron, copper products, precious metals and various other goods circulated across Eurasia. It is possible that the Siberian people had to pay iron as a tax to a powerful client such as Xianbei, as similar relations existed in the Rouran Khaganate (Bichurin, 1950); and probably during the preceding Xiongnu period (Kradin, 2011).

7. Conclusion

Interdisciplinary research conducted at the settlement of Katylyg 5 is the first step towards a refined understanding of the ancient iron production of Tuva. According to the present results, the remains of large-scale iron industry excavated at the site in the past decade date by the radiocarbon analyses to the 3rd-4th c. AD and are associated to the Kokel culture. The iron smelting furnaces operated at the site are the trapezoid in plan underground installations, which are also known in Cis-Baikal where they broadly date within the 3rd c. BCE – 10th c. AD. In Tuva, the technology of trapezoid furnaces most likely emerged after migrations from the Baikal region, linked to the impulse of westward Xianbei expansion during the 1st – 3rd c. AD.

For the first time during research on Sayan-Altai past metallurgy, the present study reveals that smelting and smithing were performed in spatially separated zones of the settlement suggesting a well-organized production pattern. The inclusions of copper (bronze) prills and the admixture of copper in iron prills in most of the analyzed smithing slags suggest that the blacksmiths were also in charge of the bronze-working. At least one of the ores smelted at the site was magnetite, associated with quartz and probably calcium carbonate.

The origin and development of iron smelting technology in Tuva still leaves many questions. Although no Xiongnu furnaces were yet securely identified in Tuva (due partly to the lack of radiocarbon analysis), finding such furnaces is likely given that this technology existed in all neighbouring regions (Altai, Khakassia and Mongolia) and Xiongnu pottery was found at one of the mining sites of Tuva (Sunchugashev, 1969: 107-108). This justifies the archaeological search for Xiongnu furnaces, and the use of integrated research approaches towards the broader investigation of ancient iron production in the region.

CRediT authorship contribution statement

Evgeny V. Vodyasov: Conceptualization, Investigation, Visualization, Project administration. **Ivan S. Stepanov:** Investigation, Visualization, Methodology, Formal analysis. **Timur R. Sadykov:** Resources, Investigation. **Evgeniya M. Asochakova:** Formal analysis. **Evgeniya S. Rabtsevich:** Formal analysis. **Olga V. Zaitceva:** Writing – original draft, Writing – review & editing. **Ivan A. Blinov:** Formal analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

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